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- (54) Title: TWO-COMPONENT GLUE SYSTEM FOR THE PRODUCTION OF LAMINATED WOOD PANELS
- (54) Bezeichnung: ZWEIKOMPONENTEN-LEIMSYSTEM FÜR DIE HERSTELLUNG VON BRETT-SCHICHTHOLZ

(57) Abstract

A two-component glue system suitable for the production of laminated wood panels consists of (a) an aqueous hardener mixture (component (a)) which contains (a1) 3 to 15 wt % of a Bronsted acid; (a2) 20 to 40 wt % of a mineral filler; (a3) 2 to 8 wt % of a thickener; (a4) 40 to 60 wt % water; and (b) a melamine resin (component (b)).

(57) Zusammenfassung

Zweikomponenten-Leimsystem, das für die Herstellung von Brett-Schichtholz (BS-Holz) geeignet ist, bestehend aus: a) einer wässerigen Härtermischung (Komponente (a)), enthaltend a1) 3 bis 15 Gew.-% einer Bronstedt-Säure, a2) 20 bis 40 Gew.-% eines mineralischen Füllstoffs, a3) 2 bis 8 Gew.-% eines Verdickungsmittels, a4) 40 bis 60 Gew.-% Wasser und b) einem Melaminharz (Komponente (b)).

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The present invention relates to a two-component adhesive system suitable for the production of glued laminated board (GL board, glulam), consisting of

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- a) an aqueous hardener mixture (component (a)) comprising
 - al) 3-15% by weight of a Bronstedt acid
- a2) 20-20% [sic] by weight of a mineral filler
 - a3) 2-8% by weight of a thickener
 - a4) 40-60% by weight of water

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and

- b) a melamine resin (component (b)).
- 20 The invention also relates to methods of producing GL board using the two-component adhesive system and to laminated board produced in this way.
- Glued laminated board (GL board), which is constructed from lay25 ers of wood which are glued together, is widely known (cf. Informationsdienst Holz, Holzleimbau, Bauen mit Brettschichtholz [German Wood Information Service, Glued Wood Structures, Building
 with Glued Laminated Board]; published by Arbeitsgemeinschaft
 Holz e.V. and Centrale Marketinggesellschaft, ISSN No.
- 30 0446-2114). These GL boards are employed predominantly as loadbearing structural components in housebuilding and the construction of commercial and public buildings.
- In this area of application, stringent requirements apply to the 35 mechanical strength of the structural components. The bonding strength must be sufficiently high even after many years, despite the action of climatic effects such as temperature change and humidity.
- 40 The adhesive systems used for the production of load-bearing structural components must, therefore, pass comprehensive tests carried out by state testing institutes in order to obtain the required suitability recommendation from the construction supervisory authorities.

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The basis for this testing is DIN 68141 in conjunction with DIN EN 301 and DIN EN 302 Parts 1 to 4. Part of the in-house

monitoring procedure of the manufacturers involves proving the quality of the structural components produced, by means of the delamination test in accordance with DIN EN 386 and DIN EN 391.

- 5 In addition to the requirements placed on the adhesive systems in respect of the service properties of the structural components produced with them, the producers of GL board desire certain properties relating to the processing of the adhesive systems. On the one hand, the initial drying time of the glues should be sufficiently long to enable layers of wood to which the glue has been applied to be processed within a certain period. On the other hand, the glued structural components should be able to be subjected to mechanical loading as soon as possible following their production. This requires the rapid curing of the glue after the glued boards have been joined together.
 - It is generally known in the art that phenol-resorcinol resins can be employed themselves [sic] for the production of GL board which can be used to produce load-bearing structural elements.
- 20 These laminated board elements, however, have the disadvantage that, because of the dark inherent color of the phenol-resorcinol resins, the glued joints in the finished structural component appears [sic] as black lines, something which is frequently rejected on esthetic grounds.
- Swedish Patent 78 10 982 discloses liquid hardeners. These are aqueous suspensions comprising, based on the aqueous suspension, 1-25% by weight of acid or acid donors, 30-90% by weight of filler, for example a mineral filler such as kaolin, 0.5-10% by weight of thickener and 0-50% by weight of a formaldehyde absorber. The hardener has a solids content of between 35 and 85 percent by weight and a viscosity of from 2000 to 12000 mPa·s at 20°C.
- 35 It is recommended that the hardener be employed for curing ureaformaldehyde resins. GL board produced using these binderhardener systems, however, does not have the required strength, especially when employed for exterior applications.
- 40 It is therefore an object of the present invention to provide two-component adhesive systems for the production of GL board which exhibit no dark glue joints and which have the mechanical properties required by the construction industry.
- 45 We have found that this object is achieved by the two-component adhesive systems defined at the outset.

The hardener mixture (component (a)) preferably has a viscosity of from 3000 to 10,000 mPa \cdot s, particularly preferably from 4000 to 8000 mPa \cdot s, based on a measurement temperature of 20°C and a shearing rate of 30 s⁻¹.

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The hardener mixture preferably comprises

al) 5-12%, particularly 7-10%, by weight of a Bronstedt acid

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- 10 a2) 20-40%, particularly preferably 25-35%, by weight of a mineral filler
 - a3) 2-8%, particularly preferably 4-6%, by weight of a thickener
- 15 a4) 35-60%, particularly preferably 40-55%, by weight of water.

Examples of suitable Bronstedt acids are mineral acids such as phosphoric acid or organic acids, for example acetic acid, propionic acid, oxalic acid or, in particular, formic acid.

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Particularly suitable mineral fillers are kaolin or other aluminosilicates having a layer structure.

Suitable thickeners are polyvinyl alcohol or partially hydrolyzed polyvinyl acetate or mixtures thereof. Polyvinyl alcohol is commercially available, for example, under the name Mowiol® (from Hoechst). In the case of partially hydrolyzed polyvinyl acetate, at least 50%, preferably 80%, of the ester groups originally present in the polyvinyl acetate have been hydrolyzed. Component 30 (c) [sic] is used in amounts of 1-20% by weight, preferably 1-8% by weight.

In addition to the mineral filler the hardener mixture can comprise an organic filler. The latter generally comprises fillers which are commonly present in wood glues, preferably in the form of fibers, examples being cellulose or wood flour.

The organic filler is usually present in component (a) in amounts of 10-30% by weight, based on the mineral filler.

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Furthermore, the hardener mixture advantageously comprises 1-20%, particularly preferably 3-8%, by weight of resorcinol, based on the overall amount of components (al) to (a4), if GL board is desired which is particularly resistant to the effects of moisture.

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In addition, customary auxiliaries and additives, such as antifoams or emulsifiers, can also be added to the hardener mixture, in an amount of up to 3% by weight based on the mixing components (al) to (a4).

In the case of component (b), the melamine resin, suitable com-5 mercially available resins are those known, for example, from Ullmanns Encyklopädie der technischen Chemie, 4th edition, volume 7, pp. 408 and 409.

These are preferably melamine resins comprising

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- bl) melamine
- b2) 2.1-8.4 mol, preferably 2.5-4.6 mol, of formaldehyde per mole of melamine

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- b3) 0-10 mol, preferably 0-0.5 mol, per mole of formaldehyde of a further compound which is capable of reacting with formaldehyde in a polycondensation or polyaddition reaction.
- 20 The melamine (component bl) is generally employed in solid form.

The formaldehyde (component b2) is generally employed in the form of a 30-50% strength by weight solution or as paraformaldehyde.

- 25 Particularly suitable components (b3) are those employed together with formaldehyde in the preparation of amino resins (cf. Ull-manns Encyklopädie der technischen Chemie, 4th edition, volume 7, pp. 403-422), examples thus being urea, dicyandiamide and guanamines such as benzoguanamine and acetoguanamine. Bisquanamines
- 30 [sic] such as adipo-, glutaro- and methylolglutarobisguanamine, and compounds comprising more than one, for example condensed aminotriazine rings, are likewise suitable.

With particular preference, component (b) comprises

- bl) 15-30% by weight of melamine
- b2) 15-30% by weight of formaldehyde
- b3) 5-25% by weight of urea, dicyandiamide and/or quanamines.
- 40 The melamine resins are preferably prepared by the customary techniques which are known, for example, from Ullmanns Encyklopädie der technischen Chemie, 4th edition, Volume 7, pp. 403-422.
- 45 The reaction is usually carried out in aqueous solution, with the proportion of water making up 25-50% by weight based on the weight of components (b1) to (b3). The pH is generally 7-10, 5

[sic] preferably 9.0-10.0, and the reaction temperature is 60-110°C, preferably 80-110°C.

The reaction is generally carried out until an initial melamine 5 resin or elasticated melamine resin has been formed. In the form of 60% strength by weight aqueous solutions, these reaction products generally have a viscosity of 350-1200 mPa·s, preferably 450-800 mPa·s, at 20°C. Under the specified reaction conditions, this usually dictates a reaction time of 70-150 minutes.

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The reaction can be terminated by cooling the reaction mixtures to below 50°C, preferably to 20-30°C.

In general the desire is for aqueous elasticated melamine resins, 15 especially those with a solids content of 50-70% by weight. Where the melamine resins are not obtained directly with the desired solids content, the latter can be modified by distilling off water, preferably under reduced pressure, or by mixing with water.

- 20 To increase the elasticity of the adhesives, they advantageously comprise aliphatic polyols in amounts of 5-18% by weight, preferably 9-15% by weight, based on the solids content of the melamine resins. These polyols can be mixed in with the starting materials used to prepare the melamine resins or can be added to the fin-
- 25 ished melamine resin. Suitable polyols are especially C_2-C_{20} polyols, preferably diols such as ethylene glycol, diethylene glycol, dipropylene glycol and butanediol, and triols such as glycerol.

In addition, in order to improve the performance properties of 30 the adhesives, for example their reactivity and flexibility, it is possible to add modifiers such as ϵ -caprolactam and other carboxamides in amounts of 1-7% by weight, preferably 1-3% by weight, to the melamine resins, based on their solids content.

- 35 The melamine resins can additionally comprise if desired up to 20% by weight, preferably 9-15% by weight, based on the solids content, of organic fillers. Suitable organic fillers here are the same as those which can also be present in component (a).
- 40 The novel two-component adhesive system can be employed by producing a liquid adhesive mass, by mixing components (a) and (b), and applying this liquor at least to one of the two surfaces which are to be bonded by the adhesive.
- 45 The liquid adhesive masses preferably consist of components (a) and (b) in a weight ratio of 0.1:1-0.5:1, particularly preferably 0.2:1-0.4:1.

The preparation of the liquid adhesive masses from components (a) and (b) is not critical and is advantageously carried out by stirring one component into the other.

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The liquid resin masses very particularly preferably have the following composition, the constituents listed adding up to 100 parts by weight (pbw):

- 10 0.5-2.5 pbw of formic acid
 - 0.3-1.5 pbw of thickener
 - 12-25 pbw of melamine in the form of the melamine resin
 - 4-21 pbw of urea in the form of the melamine resin
 - 12-25 pbw of formaldehyde in the form of the melamine resin
- 15 4-12 pbw of organic filler
 - 3-13 pbw of C_2-C_{20} polyols
 - 0-5 pbw of modifier
 - 0-4 pbw of resorcinol
 - 3-9 pbw of mineral filler
- 20 remainder to 100 pbw: water

The novel two-component adhesive systems are particularly suitable for gluing solid pieces of wood. They are particularly suitable for producing GL board which is constructed from a plurality of, preferably 5-70, layers of wood, for example wooden boards with a thickness from 10 to 43 mm.

The layers of wood are advantageously glued by a technique which is generally known in the wood industry.

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In this context, examples of possible procedures are as follows:

Ia a liquid adhesive mass is prepared by mixing components (a)
and (b),

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IIa glued layers of wood are produced by applying the liquid adhesive mass to one or both sides of layers of wood, and

IIIathe glued layers of wood are brought into contact with one another, or with layers of wood to which no glue has been applied, in such a way that there is a layer of glue between each two layers of wood to be glued together,

with the proviso that operations Ia to IIIa are carried out with-45 in a period of 45-150 minutes, preferably 60-120 minutes,

- Ib glued layers of wood are produced by applying components (a) and (b) separately from one another to one or both sides of layers of wood, so that both component (a) and component (b) are applied to both sides or one side of the layer of wood, and
- IIb the glued layers of wood are brought into contact with one another, or with layers of wood to which no glue has been applied, in such a way that the two adhesive components are in each case between two layers of wood,

or

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15 Ic glued layers of wood are produced by applying components (a) and (b) separately from one another to one or both sides of layers of wood, so that in each case only one component of the two-component adhesive system is present on a single side of a layer of wood, and

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- IIc the glued layers of wood are brought into contact with one another in such a way that in each case both components (a) and (b) are between two layers of wood to be glued together.
- 25 In the case of separate application of components (a) and (b), the application ratio of components (a) to (b) per unit area of the layers of wood in step IIb or IIc is 0.2:1.0-0.5:1.0, preferably 0.3:1.0-0.4:1.0.
- 30 The overall amount of components (a) and (b) or of liquid resin mass which is applied to the layers of wood in steps IIb, IIc and/or Ia is usually $350-500 \text{ g/m}^2$.
- The layers of wood coated with the liquid adhesive mass or with 35 the individual components are generally brought into contact with one another in accordance with step IIIa, IIb or IIc with the application of a pressure of 0.6-1.0 N/mm². This pressure is generally maintained for the period required for the adhesive to cure, which is generally about 12-14 h at 20°C. Curing is normally undertaken at room temperature; above this, the curing time becomes correspondingly shorter.

The liquid adhesive masses are particularly notable for their usability following preparation, i.e. after mixing components (a) 45 and (b). They are also notable for advantageous rheological properties. Owing to their pseudoplasticity and thixotropy they do not run on vertical glued layers of wood and do not form threads.

In particular, the GL boards produced with the novel two-component adhesive system have outstanding mechanical properties which are retained even in exterior applications.

5 Examples

Preparation of the hardener mixtures

Hardener mixture 1

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4000 g of a 10% strength polyvinyl alcohol solution, 642 g of water, 1058 g of 85% strength formic acid, 200 g of propylene carbonate, 600 g of resorcinol, 3000 g of china clay and 500 g of cellulose fibers were intimately mixed in succession, with stirring using a high-speed stirrer, and the mixture was thoroughly

15 ring using a high-speed stirrer, and the mixture was thoroughly homogenized.

The finished hardener had the following characteristics: Density at 20°C [g/ml] 1.315

20 Viscosity at 20°C, shear 30/s [mPa·s] 4802 pH at 20°C, measured using glass electrode 1.3.

Hardener mixture 2

25 5320 g of a 10.6% strength polyvinyl alcohol solution, 239 g of water, 941 g of 85% strength formic acid, 3000 g of china clay and 500 g of cellulose fibers were intimately mixed in succession, with stirring using a high-speed stirrer, and the mixture was thoroughly homogenized.

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The finished hardener had the following characteristics:

Density at 20°C [g/ml] 1.265

Viscosity at 20°C, shear 30/s [mPa·s] 5659

pH at 20°C, measured using glass electrode 1.4.

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Preparation of the melamine resins

Melamine resin A

- 40 A mixture of 2515 g (33.5 mol) of a 40% strength formaldehyde solution, 2575 g of a urea-formaldehyde precondensate (10.7 mol of urea and 42.9 mol of formaldehyde), 300 g of water, 15 g of diethylethanolamine and 2380 g (18.9 mol) of melamine were [sic] heated to 95°C and subjected to condensation at this temperature
- 45 for 75 minutes. The mixture was then cooled to 87°C and condensation was continued to a viscosity of about 900 mPa·s (measured at 20°C). The pH was kept constant at about 9.5 throughout condensa-

tion. After cooling to 50°C, 415 g (6.9 mol) of urea were added and cooling was continued to room temperature. The viscosity of the mixture was 715 mPa·s (measured at 20°C) and it had a solids content of 62.5% by weight. 100 g of caprolactam, 600 g of ethylene glycol and 850 g of cellulose were incorporated homogeneously into this resin by stirring.

The finished resin had the following characteristics:

Viscosity

2480 mPa·s

65.2% by weight

pH

9.2

Gel time (3 g of 34% strength formic acid, 40°C) 55 minutes.

Melamine resin B

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A mixture of 2515 g (33.5 mol) of a 40% strength formaldehyde solution, 2575 g of a urea-formaldehyde precondensate (10.7 mol of urea and 42.9 mol of formaldehyde), 300 g of water, 15 g of diethylethanolamine and 2380 g (18.9 mol) of melamine were [sic] 20 heated to 93°C and subjected to condensation at this temperature for 90 minutes to a viscosity of about 600 mPa·s (measured at 20°C). The pH was kept constant throughout condensation. After cooling to 50°C, 415 g (6.9 mol) of urea were added and cooling was continued to room temperature. The viscosity of the mixture 25 was 548 mPa·s (measured at 20°C) and it had a solids content of 62.4% by weight. 850 g of diethylene glycol and 950 g of cellulose were incorporated homogeneously into this resin by stirring.

The finished resin had the following characteristics:

30 Viscosity

Solids content

PH

Gel time (3 g of 34% strength formic acid, 40°C)

58 minutes.

35 Melamine resin C

A mixture of 4550 g (60.7 mol) of a 40% strength formaldehyde solution, 350 g of a urea-formaldehyde precondensate (1.5 mol of urea and 5.8 mol of formaldehyde), 15 g of diethylethanolamine and 2700 g (21.4 mol) of melamine were [sic] heated to 97°C and subjected to condensation at this temperature for 90 minutes to a viscosity of about 580 mPa·s (measured at 20°C). The pH was kept constant throughout condensation. After cooling to 50°C, 438 g 7.3 mol) of urea were added and cooling was continued to room temperature. The viscosity of the mixture was 450 mPa·s (measured at 20°C) and it had a solids content of 59.1% by weight. 200 g of caprolactam, 300 g of 1,4-butanediol, 500 g of ethylene glycol and

947 g of cellulose were incorporated homogeneously into this resin by stirring.

The finished resin had the following characteristics:

5 Viscosity Solids content 2270 mPa·s

63.5% by weight

рН

9.4

Gel time (3 g of 34% strength formic acid, 40°C) 42 minutes.

10 The physical properties of the hardener mixtures and of the melamine resins were determined as follows:

The viscosities were measured in accordance with DIN 53019 using a rotary viscometer at 20°C. The solids contents were measured in 15 accordance with DIN 12605, weighing out 1 g of the substance into a flat glass weighing boat with a diameter of 3.5 cm and drying it in an oven at 120°C for 2 h.

The pH was measured in accordance with DIN 53785/ISO 1148.

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To measure the gel time, 100 g of melamine resin were mixed with 3 g of 34% strength formic acid and the mixture was heated to 40°C. The gel time is the time from the addition of the hardener until the sample becomes solid.

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Performance tests

For these tests a homogeneously mixed, ready-to-use liquid adhesive mass was produced from 100 parts by weight of melamine resin 30 and 20 parts by weight of hardener mixture. The tests were carried out in accordance with DIN 68141, October 1969 issue, and DIN EN 391, November 1990 issue in conjunction with DIN EN 386, November 1990 issue. The following specific parameters were tested:

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Test I Pot life, DIN 68141 Section 2.1.2

Test II Open initial drying time, DIN 68141 Section 2.1.3

- 40 Test III Effect of joint thickness and different storage sequences on bond strength, DIN 68141 Sections 2.1.1 (A) and 2.2.3 (B)
- Test IV Effect of local climatic conditions on setting rate,
 DIN 68141 Section 2.3 a) after 24 hours

Test V Delamination resistance, DIN pr EN 386

Test VI Optical assessment of the color of glue joints and their afterdarkening properties.

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The tests were carried out on the following liquid adhesive masses:

Liquid adhesive mass 1

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Melamine resin A with hardener mixture 1

Liquid adhesive mass 2

15 Melamine resin C with hardener mixture 2

Liquid adhesive mass 3

Commercial phenol-resorcinol-formaldehyde resin (Kauresin® 20 Adhesive 460 liquid from BASF AG) with a commercial hardener for phenolic resins based on paraformaldehyde and small quantities of a carboxylic acid (Kauresin® Hardener 466 powder from BASF AG).

The results of the performance tests can be seen from Tables 1 25 and 2.

Table 1:

Bond strengths (N/mm²) of bonds in accordance with DIN 68141 with 30 different joint thicknesses after storage sequences DIN 53254, January 1987 issue

35	Liquid adhesive mass	Test III A Storage sequence			Test III B Storage sequence			Test IV
		1	13	14	1	13	14	
	1	10.16	6.67	10.14	8.90	6.21	7.84	6.26
	2	10.39	7.08	9.38	8.40	6.22	6.91	5.26
40	3	11.30	8.11	10.94	11.22	6.84	9.02	5.24

Table 2:

Results of the tests for pot life and open initial drying time in accordance with DIN 68141 and of delamination in accordance with 5 DIN pr EN 391 and 386 and of the optical assessment of the glued joint color

10	Liquid adhesive mass	Pot life (min) 20°C/65% RH	Open initial drying time (min), application of 400 g/m ²			mination r 3 days	Optical assessment
15			Beech	Pine	Total joint %	Individual joint %	
	1	105	120	130	2.4	10.4	light colored glued joint, minor after-darkening
20	2	120	167	150	0		light colored glued joint, no after-darkening
	3	235	135	135	0	. 0	dark red-brown

Claims

- A two-component adhesive system suitable for the production
 of glued laminated board (GL board), consisting of
 - an aqueous hardener mixture (component (a)) comprising
 - al) 3-15% by weight of a Bronstedt acid

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- a2) 20-40% by weight of a mineral filler
- a3) 2-8% by weight of a thickener
- a4) 40-60% by weight of water

and

b) a melamine resin (component (b)).

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- 2. A two-component adhesive system as claimed in claim 1, wherein the aqueous hardener mixture (a) additionally comprises an organic filler.
- 25 3. A two-component adhesive system as claimed in claim 1 or 2, wherein component (a) additionally comprises resorcinol.
- 4. A two-component adhesive system as claimed in any of claims 1 to 3, comprising a component (b) comprising
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bl) melamine

- b2) 2.1-8.4 mol of formaldehyde per mole of melamine
- b3) 0-1.0 mol per mole of formaldehyde of a further compound which is capable of reacting with formaldehyde in a polycondensation or polyaddition reaction.
- 5. A process for producing GL board constructed from layers of wood using a two-component adhesive system as claimed in any of claims 1 to 4, wherein
 - la a liquid adhesive mass is prepared by mixing components
 (a) and (b),

- IIa glued layers of wood are produced by applying the liquid adhesive mass to one or both sides of layers of wood, and
- IIIathe glued layers of wood are brought into contact with one another, or with layers of wood to which no glue has been applied, in such a way that there is a layer of glue between each two layers of wood to be glued together,
- with the proviso that operations Ia to IIIa are carried out within a period of 45-150 minutes.
 - 6. A process for producing GL board using a two-component adhesive system as claimed in any of claims 1 to 4, wherein
- 15 Ib glued layers of wood are produced by applying components
 (a) and (b) separately from one another to one or both
 sides of layers of wood, so that both component (a) and
 component (b) are applied to both sides or one side of
 the layer of wood, and
- IIb the glued layers of wood are brought into contact with one another, or with layers of wood to which no glue has been applied, in such a way that the two adhesive components are in each case between two layers of wood.
 - 7. A process for producing GL board using a two-component adhesive system as claimed in any of claims 1 to 4, wherein
- Ic glued layers of wood are produced by applying components

 (a) and (b) separately from one another to one or both sides of layers of wood, so that in each case only one component of the two-component adhesive system is present on a single side of a layer of wood, and
- IIc the glued layers of wood are brought into contact with one another in such a way that in each case both components (a) and (b) are between two layers of wood to be glued together.
- 40 8. GL board obtainable by a process as claimed in any of claims 5 to 7.